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(54) Equipment and Process to Secure Oil, Gas, and  
By-Products from Pyrobituminous Shale and Other Matter  
Impregnated with Hydrocarbons

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ABSTRACT OF THE DISCLOSURE

The process for obtaining oil, gas and other products from pyrobituminous shales and the like includes introducing crushed shale into a retort and contacting the crushed shale in the top portion of the retort with a stream of retort gases. Hot gases are injected at an intermediate point of the retort and a stream of cold gases are injected at the bottom of the retort. The gaseous retorted matter above the zone where the shale undergoes pyrolysis is removed and directed to a cyclone for separation of heavy liquid components from a gaseous stream. The gaseous stream is then purified and compressed with a portion of the compressed stream being heated and reinjected as the stream of hot gases into the retort. The other portion of the compressed stream of gases is cooled and a liquid component consisting primarily of water and heavy oil is separated therefrom in a spray tower. The water portion is separated from the oil and recirculated to the spray tower while a part of the oily portion is separated for reuse outside the process with the remainder being recycled to the cyclone. A portion of the compressed gases from the cyclone is cooled and injected into the bottom of the retort as the stream of cold gases.

3

APPARATUS AND PROCESS FOR OBTAINING OIL, GAS  
AND BY-PRODUCTS FROM PYROBITUMINOUS SHALE  
AND OTHER MATERIAL IMPREGNATED WITH HYDROCARBONS

This invention relates to a process for producing mineral oil and other by-products from solid material, particularly pyrobituminous shale, by means of an integrated process under which the chief operation is that of retorting, substantially in the absence of air, material in the form of particles of a given size range.

The main object of this invention is to obtain liquid and gaseous hydrocarbons substantially useful as fuels and also to recover products which will be employed as sources for by-products other than those directly produced from the above mentioned retorting, after undergoing treatment which will be specified later.

Another principal object of this invention is to provide an integrated process under which energy and mass balances are optimized, so that the operation as a whole is as cheap as possible.

A main characteristic of the whole process is that the only source of raw material introduced into the system is the pyrobituminous shale or like material which is being treated, and that the circulating fluids (which act as heat exchange medium for drawing products into the retorting vessel, into the several pipes and into intermediate product treatment stations), are derived from the aforesaid raw material after it has been treated within the retorting vessel, without letting in any outside air or any other inert fluid or auxiliary reagent, other than the products derived from the retorting.

The present application also aims to improve on the process and apparatus described in our Brazilian patent No. 7105857 (and in U.S. Patent No. 3,887,453, dated June 3, 1975, which corresponds); in particular the present process is to be cheaper as regards use of energy and operating methods.

Accordingly one aspect of the present invention provides apparatus



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for securing oil, gas and by-products from material impregnated with hydrocarbons, comprising a pyrolysis retort; sealing means to maintain the retorting gases within the retort during charging of the retort with particulate material to be pyrolysed; discharge means for removing pyrolysed particles from the bottom of the retort substantially without discharge of the retorting gases; means for injecting hot retorting gas into the retort for contact with the particulate solid material in the retort for pyrolysis thereof; means for introducing colder gas into the retort at a location below the hot gas introduction means; gas outlet means from the retort and leading to separating means for separating dust and released hydrocarbons and by-products from the gas leaving the retort; means for returning a first stream of gas from said separating means to the retort by way of said hot gas injector means; means for returning a second stream of said gas from the separating means to said colder gas injection means in the retort by way of a heat recovery unit; and means for directing a third stream of the separated gases from said retort to a location of use of the gases; wherein the said hot gas injector means comprise a bundle of mutually parallel pipes of polygonal cross-section comprising an upper vertex defined by the angle of intersection of two roof plates which are joined to two parallel vertical side plates each joined at their lower ends to a floor of the associated pipe, each of said side plates including a row of gas discharge holes along the length of the pipe, in the upper portion of said side wall, the lower portion of said roof plate being extended downwardly and outwardly beyond the said line of intersection with the said plate to create an overhang for protecting the gas discharge holes from impact by descending solid particles.

Another aspect of the present invention provides a process for recovering oil, gas and by-products thereof from material impregnated with hydrocarbons, comprising introducing the hydrocarbon-impregnated

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material to a retort while containing the retort gas within the retort and preventing ingress of oxygen into the retort with the incoming particulate material; introducing hot pyrolyzing gas into the retort for contact with the particulate hydrocarbon-impregnated material therewithin; introducing a relatively colder gas into the retort below the level of introduction of the hot pyrolyzing gas; discharging the pyrolyzed particulate material from the base of the retort while excluding discharge of the retort gases with said discharge material; discharging the retort gases and separating therefrom dust and hydrocarbon mist discharged therewith; recovering the separated hydrocarbons from said separating system; passing the gas bearing residual hydrocarbon mist and dust to a spray scrubbing tower; spraying water into the spray scrubbing tower to wash hydrocarbons and dust from the gas which is then discharged to atmosphere; separating the water and hydrocarbon discharged from the spray scrubbing tower in first and second separating and settling stages in which the first separating and settling stage provides water for the spray-scrubbing operation and a hydrocarbon constituent which is further separated in the second separating-settling stage; dividing the separated hydrocarbons and dust from the second separating-settling stage into a first stream which is returned to the retort gas separating operation and a second stream which is delivered for commercial use; cooling the gaseous phase of the retort gas separating operation before separation into said first and second streams, and further cooling the said second stream before return to the retort.

The process described herein can be used on any solid material which provides oil upon being heated, preferably pyrobituminous shale, and for the sake of economy the oil content of the shale charge should not be less than 4% by weight, in the dry state.

Before being subjected to the processing cycle, the shale should be crushed down to a charge range of from 0.32cm to 15.24cm rated particle size, preferably from 0.64cm to 7.62cm.

In order that the present invention may be better understood, the following description is given, merely by way of example, with reference to the accompanying drawings, in which:-

Figure 1 is a schematic representation of the apparatus involved in carrying out the process;

Figure 2 is a part sectional view of the charging mechanism and upper seal for the processing plant of this invention;

Figure 3 is a part sectional view from above, of a rotating seal mechanism shown in Figure 2;

Figure 4 is a longitudinal section of the non-segregating auxiliary conveying mechanism which is part of the plant shown in schematic view in Figure 1;

Figure 5 shows the arrangement of the device for injecting hot gases into the retort;

Figure 5a is a schematic top plan view of the set of hot gas injection ducts showing how they fit into the walls of the retort;

Figure 6 is a partial section, taken on a horizontal plane, of the controlled discharge mechanism for solids which lies in the bottom of the retort shown in Figure 1;

Figure 7 is a side sectional view of the device shown in Figure 6;

Figure 8 is a considerably simplified top plan view of the mechanism shown in Figures 6 and 7 meant to reveal certain details thereof; and

Figure 9 is a schematic representation of a gas injector nozzle in the bottom of the retort shown in Figure 1.

-5-

Referring first to Figure 1, it can be seen that the charge 1 of shale or other like solid material which is to be treated by the apparatus to be described is, after being suitably crushed, taken to a hopper 2 which is provided in its bottom with a deflecting valve (not shown in the drawing) to enable the descending flow of crushed solids to run through either one of two downwardly sloping ducts 3 leading to one of the charging and sealing mechanisms 4. A rotating seal of the charging and sealing mechanism 4 is shown schematically in part sectional view in Figure 2 and in top plan in Figure 3.

The rotating seal 4 consists chiefly of a closed cylindrical housing or frame 410 provided with an inlet opening 411 in its top cover 412 and an outlet opening 416 in its bottom cover 413, such cylindrical housing 410 having a rotatable shaft 414 running from the middle of the top cover 412 to the middle of its bottom cover 413 and carrying radially extending vanes 415 (Figure 3) the number of which may vary according to the particle size of the material flow or to its rate of flow. In this case there are eight vanes 415 symmetrically arranged and fixed around the shaft 414. Such vanes 415 have their outermost ends fixed to a cylindrical shell, thereby comprising a rotor in the form of a regular body 417. It should be noted that, particularly in the example shown in Figures 2 and 3, the vanes 415 are rectangular in shape, so that as the rotor turns, the vanes sweep all the inside of such cylindrical body 410 during rotation of the vertical central shaft to which the vanes are fixed. It should be added that the vertical shaft 414 is driven by an external drive source (not specifically described nor shown in the drawings). Another important feature of the rotating seal 4 described herein by way of example, is that the inlet opening 411 in the top cover lies diametrically opposite the outlet opening 416 in the bottom cover 413, the inlet at the top being joined to the sloping duct (Figure 1) whereas the outlet opening 416 in the bottom is joined to the

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vertical duct 5 into which the solid particles entering the inlet opening 411 will fall, so that the particles will be swept by the vanes 415 of the rotor 417 to be discharged through the outlet 416 in the bottom panel 413. In the preferred example the vertical duct 5 is provided, at a given point somewhere along it, with an injected stream of suitable gaseous fluid, by convenient means (represented for example in Figure 3 as a pipe 510). The gaseous fluid might be steam or an inert gas (preferably the latter), for pressurising not only the duct 5 but also the inside of the rotating seal 4 and another similar rotating seal 6 at the bottom end of the vertical duct 5. This excludes air from being drawn into the upper seal 4 with the solid particles, thus preventing any oxygen from entering the retorting system and the lower seal 6; and also prevents any retort gases from rising up the duct 5 into said rotating seal 6.

It should be explained that the relative positioning and number of the inlet opening 411 and the outlet opening 416 in the cylindrical body 410 of the rotating seal 4 disclosed herein must not be taken as essential, as the description provided above is merely meant as an example to aid understanding of the arrangement.

As is to be understood from the foregoing description, the vertical duct 5 connects the upper seal 4 to the lower one 6, which may itself either be directly connected to some other mechanism, for instance, a non-segregating particle distributing mechanism 8 which leads straight into the retort 9, or be provided with another vertical duct 7, like the duct 5 which leads into the top of another rotating seal like the ones already referred to. This vertical arrangement may be repeated as many times as needed in order to ensure sealing in special cases.

In the present example, and since it has been found to be practical in several cases, pairs of rotating seals 4,6 joined by one duct 5 have

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-7-

proved to be efficient. As can be seen from Figure 1, there are two charging and sealing mechanisms, so that one can be run while the other is being serviced.

It should be added that the lower seal 6 can be identical to the seal 4, having top and bottom covers 610 and 611, a rotating shaft 612 which can be joined to a shaft 414 of the upper seal 4 and inlet and outlet openings 613 and 614, and a vaned rotor as described above.

It is an important feature of the construction of the covers of the rotating seals and also of the axially inwardly facing edges of the rotors, that they are provided with special non-abrading coverings, which are releasable parts so fastened as to enable them to be removed and changed at maintenance periods.

Thus the shale or other solid crushed matter to be treated, after having been led to the hopper 2, travels along one of the sloping ducts 3 to the charging and sealing mechanism provided with its rotating seals 4 and 6 joined by a vertical duct 5 slightly pressurized by an inert gas, from where it will flow by gravity to the non-segregating solids distribution mechanism 8, and then to the body of the retort 9, where it will undergo actual chemical and physio-chemical retorting stages.

Actually, considering the apparatus as a whole, the non-segregating mechanism below outlet pipe 7, which connects the outlet opening of the lower rotating seal 6 to said non-segregating distribution mechanism arrangement 8, lies within the top housing of the retort vessel. However, because of the several stages of the operation which take place in each part, and even though there are no very clear boundary lines, some breakdown will now be made in the description in order to try to make it clearer.

The non-segregating mechanism 8 is shown in greater detail in Figure 4, which shows a united set of interdependent parts; it has been

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shown broken down into areas I, II, III and IV in terms of the parts that make up each of these areas.

Thus, area I depicts the cylindrical housing 809 which surrounds a rotating distributor 803 and is a funnel-shaped part, whose wider top opening lies immediately below the top cover 802 of area I, which encompasses openings 801 into which the ducts 7 carrying the granulated solid particles coming from rotating seal 6 open. The funnel-shaped rotating distributor ends at its bottom in a narrow pipe 808 and is fastened to a shaft 806 which is supported by a bearing 807 at which it is slowly rotated by way of a motor 804 coupled to the shaft 806 by means of a reduction gear 805.

The solids discharged into the funnel-shaped rotating distributor 803 fall from it, clear of surrounding shaft 818, into area II and are led over the funnel-shaped separating wall 812 to gather inside portions 816 and 817 bounded by (a) the outside wall 809 of the plant, (b) the funnel-shaped separating wall 812 and (c) the innermost wall 810 of the inside conical piece which runs upwards of the fixed shaft 818, while undergoing a minimum of segregation by particle size.

From area II the solids continue to flow under gravity along the descending ducts 813 which make up area III and lead into area IV, which is the top part of the retort 9 proper. Together the position of the narrower bottom piping 808 of the funnel-shaped distributor relative to the funnel-shaped separating wall 812, plus the length and slope of the descending ducts 813 in area III, not only provide reduced segregation of different size particles but also effect a considerable reduction in the formation of "valleys" 814 in area IV.

"Valleys" is the name given to dips in the surface of the mass of particles at rest, caused by the uneven build up thereof.

The body of the retort 9 itself is cylindrical in shape, and is

internally lined with special refractory material which not only cuts down on heat exchange with the outside but also protects the inside of the retort wall against erosion by the friction of the downwardly moving solid particles. Naturally, since this is a reactor which must be well thermally insulated, the body of the retort must as far as possible be provided with an external lagging of various materials well known in the art.

Starting from the top of the retort and working down to the bottom thereof, without however once again going into detail about the already described non-segregating charging mechanism, the following important retorting features will be described at greater length, as necessary, in order to give a better understanding of the invention:-

(a) at the point where the descending ducts of the non-segregating mechanism lie, the retort is provided with either one opening to which an outlet duct 10 is attached, or many openings connected to outlet ducts which at some point outside the retort join up with a common duct for a gaseous flow containing (in the form of steam and/or mist) the liquid fraction created by the retorting operation, and also finely divided solids drawn entrained by said gaseous flow.

(b) at an intermediate point, between the bottom end of the downward ducts 813 of the non-segregating charging mechanism and the bottom of the retort, lies a set 11 of hot gas injectors which will be described in greater detail and are shown schematically in Figure 5. It should however be pointed out that the exact location of such injectors will depend in each case upon the final retort design as drawn up by the process engineering specialists, since it will depend on such factors as the diameter of the retort, and the upward speed of the gases, which in turn will depend on the loss of charge in the descending bed of solids.

(c) at a point in the bottom of the cylindrical body where the retort 9 begins to reduce in diameter, and become funnel-shaped, is a discharging

-10-

mechanism 13, to be described in greater detail below with reference to Figures 5, 6 and 7.

(d) at the conical body 14, which is a downward extension of the cylindrical body of the retort 9 and is slightly below the discharging mechanism 13, are holes arranged horizontally around the conical body, to which are fitted cold retorting gas injection nozzles 15 which are connected by non-illustrated piping to a cold gas conduction duct at some point of the by-product treatment system to be described later.

Figure 5 shows that the set of hot gas injectors shown in Figure 1 is largely made up of elongate prismatic ducts 111 of irregular hexagon cross-section. The number and arrangement of such injectors 111 is strategically worked out within the descending bed of granulated solids inside the cylindrical body of the retort 9. This hexagonal design is a result of technical factors connected with the flow properties of granulated solids. It is obvious to those skilled in the art that Figure 5 represents the set of injectors merely schematically, since it is not necessary to draw up any precise arrangement details for such injectors 111 inside the retort. The expert will readily appreciate that to anyone looking at the front of the set, the faces of the injectors represented by front plates 116 would not appear to be lined up as shown schematically in Figure 5.

Before describing the hot gas injection system in further detail, it will be compared with the system disclosed in Brazilian Patent 7105857 so that the new approach as will be disclosed later provides an astonishing saving in cost of operation particularly as regards improved use of heat and higher yield derived from the output.

In the equipment described in Brazilian Patent 7105857 (at page 4, line 32; page 5, lines 1 to 3; page 5, lines 28 to 32; and page 6, lines 1 to 3), the hot gases were introduced by means of circular cross-section pipes,

provided with two lines of holes pointing downwardly at an angle of 15° to either side of the vertical and with each jet about 90° displaced from the next. To protect the pipes and holes each gas injection pipe was topped by a straight piece of right angle profile with the fold-edge uppermost which acted as a covering ridge to prevent any abrading of such piping by the moving of solid particles. However, in spite of the tendency of hot gases to spread out among the particles of the descending bed because of the pressure at which they were injected, there was still (between the protecting right angle profile and the pipe) a dead space which was devoid of any solids to be sought out by the hot gases, and this led to an irregular distribution of heat to the solids. It should be pointed out that achieving a preferred path for gases in any treatment process involving a moving bed is one of the most difficult design aspects, particularly as regards increasing the yield of the process concerned.

However, in the present design of hot gas distributor 11, all the problems of the prior art have been overcome by the introduction of the novel aspects to be described below:-

(i) the right angle profile protection has been done away with and therefore there is no longer a dead space empty of solids in the middle of the descending bed of solids, the shape of the cross-section of each duct now being an irregular hexagon;

(ii) the side walls 114, shown in Figure 5 as seen merely from the right side of each of the prisms that make up the injectors 111, are provided with one or several rows of holes 115 along the entire length of the mutually parallel vertical side walls 114;

(iii) in one preferred embodiment the arrangement of the row of holes 115 in the walls 114 is near the top, but just slightly below the line at which the cover plates 112 meet the vertical side walls 114. One

embodiment of the ducts 111 may involve a slight extension of the cover plates 112 downwardly beyond the line where they meet the vertical side walls 114 to create overhangs for protecting such holes 115 from being struck by descending solids. Another advantage of having the row of holes 115 in a top part of the vertical side walls 114 is that it prevents any hot gases which might be introduced at a point lower down the walls 114, and which could meet another gaseous stream from the opposite wall of the neighbouring duct 111, from creating a turbulent gas cushion which could affect proper downward flow of solid particles. Practice has shown that distribution of the gaseous jet at an upper spot on the side walls 114 enables rapid dispersion of the gases into the mass of descending solids without in any way hindering the solids flow;

(iv) as in the case of the top walls 112, the bottom walls 113 are made of extended rectangular plates joined to one another side to side to create a bottom vertex;

(v) the front part is made up of the blind part 116 of irregular hexagon shape.

It should be explained that the preferred angles for the vertices of the top walls of the cover 112 and the bottom walls 113 depend on the effect caused by the flow of the bed of solids crushed into particles, whose diameter may range from 0.32 cm to 15.24 cm so as to enable hot gas injection ducts to provide an abundant, uniform, and efficient distribution of the gases without affecting the flow of such solids.

Furthermore the arrangement of the holes 115 in the side walls of each prismatic duct, as described above, means that the hot gases are directly injected into the descending bed without need for any baffles which might lead to further loss of charge and without any turbulence in the gaseous flow beyond that usually caused by the gases striking the solid particles, and without any need to incline the gaseous jet.

-13-

Also the proposed injecting device has the advantage of enabling the difference between the pressure inside each prismatic duct 111 and that in the descending bed of solids to be controlled, since the whole of the inside of said ducts has been designed to hold a considerable volume of gas under pressure which will be made to flow in terms of a planned arrangement of holes whose diameter and spacing will depend on the speed of the gases within the bed, the temperature of the charge, and the loss of charge, together with the rate of flow of the solids and the particle size and also on the diameter of the retort 9.

It has been found that the diameter of the holes 115 and their number are of interest to this invention, and that in addition to depending on the temperature, pressure and particle size of the solids as already stated, the diameter and number of holes also depend on the difference in the permitted rate of discharge between the first and last holes, which rate should be in the range of from 1 to 5%, preferably from 2 to 5%, in order to keep a balance between the heat requirements of the process and the cost of circulating the gases (through compressors, intermediate pumps and control circuits). The distance between the injector ducts 111 should be less than 2.5 times the width of such duct and more than 4 times the diameter of the largest solid particle in the bed.

With further reference to Figure 5, it should be understood that all the ducts which carry the hot gas for discharge out of the side holes 115, lead from the heater 44 and along the duct 45 to join up with a manifold 119 (shown in Figure 5a) having several nozzles which, whether within or outside the retort 9, are joined to the inlet of the respective injection ducts 111. Though in some embodiments it may be preferred that these nozzles of the hot gas manifold 119 should lie outside the retort 9 this should not be regarded as essential to the present invention. Likewise there is no need for the flow direction of incoming hot gases to be always the same in

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all the prismatic ducts, for distribution thereof may be made to alternate according to the engineering or cost aspects of each design.

In an alternative embodiment of this invention the blind wall 116 may have a rectangular form (as at 118 in Figure 5) and extend slightly beyond the end of the injection duct 111 to make it easier for the wall 116 to support the injection ducts 111 in slots in the retort walls.

Figure 5a is a schematic view from above of a set 11 of hot gas injection ducts 111 showing the ducts entering the retort through the walls 26C of the retort 9 after leaving the distribution pipe 119 outside the retort and also showing how the end stretch of each injection duct 111 rests on a boss on the opposite retort walls intended to support it, and how such bosses are formed as a deformation of the retort walls on which they lie.

It should be pointed out however that this design feature does not affect the function of the hot gas injection ducts 111. Other designs may be followed; for example, the slots 120 in the retort wall may be hexagonal, or even rectangular, in shape to take the supporting parts of the hexagonally-shaped end portion of each injection duct 111.

As referred to above, next to the cylindrical bottom part of the retort 9 and inside it is the discharge mechanism 13, as can be easily seen in the partly cutaway plan view of Figure 6 and in Figure 7 which is a cross-section of merely half of the set of components.

Thus discharge mechanism 13 consists basically of two sets A and B of stationary parts, and of moving set C, details of which are evident from Figures 6 and 7.

Only a limited number of elements making up the discharge mechanism is shown, for ease of understanding. It should however be understood that this number is not a positive limitation, for the number is

-15-

always a function of the diameter of the retort 9 and of the size of the solid particles which are to undergo processing.

The set A is made up of what are termed herein "retaining tables", which are flat plates 1A, 2A, 3A, 4A cut in the shape of circular annuli spaced apart in the same plane concentrically within the retort 9 next to the bottom of the cylindrical body thereof. These annuli are concentric to the retort wall and rest on, and are kept rigidly together as a set by, suitable means, such as slender but sturdy girders which in turn rest firmly on the retort walls 26C and hold up that set, in addition to enabling its surfaces to remain free and as horizontal and as flat as is possible.

In another design the retaining tables may be mounted upon a frame of tubular or "box" girders assembled in a lattice arrangement but in such a way as hardly to interfere at all with the flow of solids.

The spaces between each such circular annulus 1A, 2A, 3A or 4A and that right next to it are also circular annuli through which flow the solids due to be discharged.

Such annular spaces are covered by baffles 11B, 12B, 13B, 14B, 15B, over-hanging the plane of the free surface of the retaining tables at a spacing which must be greater than the largest size of descending solid particle, in such a way that, looking downward from above as shown in Figure 6, the spaces 20A, 21A, 22A, 23A, 24A are wholly covered by such baffles 11B, 12B, 13B, 14B, 15B. It should be noted however that, because of its position, the central empty space 24A is not an annulus but just a circle. Each of these baffles is in the shape of a ring made up of two curved plates arranged at an angle to the horizontal in such a way that, as is to be seen from Figure 7, if one of the rings making up the baffles were to be cut through, the profile would be that of an isosceles triangle, or (in another design thereof) just two sides thereof would be at an obtuse angle

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-16-

(if there were no base plate for said baffles). In Figure 7 the baffles appear as isosceles triangles as is preferred in accordance with the invention. We will also see that because of its position, the central baffle 15B in both Figure 6 and Figure 7 is not really a ring but rather a cone meant to cover the central circle 24A of the above-mentioned set of "retaining tables". It should also be noted that baffle 14B has a profile which is not really a triangle but rather an irregular trapezium, since one of its faces stands directly upon the retort wall 26C as shown in Figure 7. A further important feature of the set is the compensating baffles only two of which, 16B, 17B, are illustrated, merely to show their position in relation to the centre of the set of retaining tables and in relation to the other circular baffles already described. As can be seen from Figure 6, the compensating baffles 16B, 17B link up the concentric circular baffles; their relative arrangement, provided merely as an example, is shown in Figure 8.

If it be considered that the cylindrical body of the retort is practically full of solid particles which are undergoing pyrolysis, as described later, it will be seen that the configuration of the bed of solids at rest within the controlled discharge mechanism is as follows: the solid particles fall upon the retaining tables 1A, 2A, 3A, 4A towards which they are deflected by the baffles 11B, 12B, 13B, 14B, 15B and the several compensating baffles 16B, 17B.

Considering now a working representation of the discharge mechanism 13, we see that the final aim is to cause the solids gathered on the "retaining tables" to fall into area 14 which in the example represented in Figure 1 is funnel-shaped body as an inverted truncated cone which extends downwards as a descending duct 16 to end up at the final rejection mechanism 17 for the retorted solids, as will be shown later.

In order to cause this controlled drop of the solid particles off the

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retaining tables 1A, 2A, 3A, 4A a set of scrapers "C" is provided as the moving apart of the controlled discharge mechanism, the description and operation of which will now be given in greater detail.

The set C of scrapers consists chiefly of metal scraper rings 5C, 6C, 7C, 8C whose diameter is such that when lying at rest upon the retaining tables 1A, 2A, 3A, 4A respectively, they lie about half-way between the edges of each of the retaining tables, it being supposed that the radially extending parts 9C supporting such scraper rings 5C, 6C, 7C, 8C in this rest position converge towards a common point of intersection which coincides with the geometrical centre of the set of concentric retaining tables and the set of concentric baffles. In the sectional view of Figure 7 the scraper rings are shown with a rectangular profile, and a height less than the distance between the bottom edge of the concentric baffles and the plane of the top free surface of the retaining tables, and preferably greater than the size of the largest particle flowing through the discharge mechanism 13.

In a preferred design the radially extending parts 9C, shown in section, have a circular profile and as represented in Figure 8, extend radially beyond the walls of the cylindrical portion of the retort 9, so that at each outer end of such radial parts 9C a hydraulic drive 19C is coupled. A piston of the hydraulic drive drives the stem which pushes its respective extended radial part 9C which, being joined to the other supporting parts 9C of the scraper rings 5C, 6C, 7C, 8C, causes the scraper rings to move, thereby shifting the solids gathered on the retaining tables 1A, 2A, 3A, 4A into the spaces 20A, 21A, 22A, 23A, 24A from where they drop into the bottom region 14 of the retorting vessel. It must be mentioned however that since the end of every extended part 9C is provided with a hydraulic drive 19C the set of scrapers will move in a given direction if only one of the hydraulic drives 19C is activated, while, depending to the design, it

may be decided to balance forces by simultaneously advancing that hydraulic drive 19C which is diametrically opposite to one that is retracting. Thus, given that there is an alternating action of the various hydraulic drives 19C, it will be easily understood by those skilled in the art that the overall movement of the scrapers will describe a regular polygon (as defined by the movement of a given reference point over the set of scrapers) to ensure that the whole area of the retaining tables is swept by the scraper rings, and therefore that the flow of solids is as even as possible.

As can be seen from Figure 7, where the reciprocating radial extrusion 9C passes through the wall 26C of the retort 9 it is provided with a retainer 10C which prevents escape of the retort gases. For the same purpose as well as keeping a certain internal pressure within it, the retainer 10C includes means 18C for the injection of an inert gas into it, and thus this retainer pressurizing gas is also injected into the retort 9. In practice the pressurizing gas that also enters the retort is the cold recycle gas as will be explained later.

It is easy to understand that the aim of the programmed operation of the discharge mechanism is to time the hydraulic drives 19C so that the solid particles will be afforded optimum residence time throughout the whole cross-section of the retort.

As stated above, the solid particles that have undergone treatment within the retort 9 are discharged by the discharge mechanism 13 into the area 14 from which they will slide towards the rejection mechanism 17 for retorted solids down a descending duct 16 which operates as a water bath building up a water column that reaches a pre-established level inside it and that provides a seal for the interior of the retorting apparatus.

At a given point in the funnel-shaped region, more precisely at a point below the discharge mechanism 13, are the injection nozzles 15 to inject cold gases into the bottom of the retort.

At this point the apparatus of the present application should be compared with the cold gas injection system described in Brazilian Patent 7105857 so as to illustrate the improvements of this invention because the pyrolysis treatment of granulated solids in a downward moving bed leads to a noticeably better heat balance and therefore to an improved physical and chemical process in general, particularly if the solids are pyrobituminous shales.

In the Brazilian Patent the cold gases were injected into a series of mutually parallel horizontal pipes each provided with two rows of downwardly pointing holes spaced such that the jets of gas were approximately at right-angles to one another as described above with reference to the hot gas injectors 11. That arrangement caused the gases to spread among the descending particles in order to bring about the heat exchange so that the heated material, particularly pyrobituminous shales, should drop down to the water bath in the rejection and sealing mechanism 17 with as low a temperature as possible and that the cold gases upon rising to the hot gas distributor level of the retort 11 should promptly begin to heat up to close to the temperature of the injected hot gases, at which point it is desired that the pyrolysis reaction shall have risen to its greatest intensity.

However practice has shown that to lead cold gases into a set of horizontal pipes and to force the gases out of rather narrow holes causes an unnecessary loss of charge, and did not help to bring about any rapid equalization in the heat exchange at the cold gas injection level.

In this application the cold gases are injected through tubular nozzles 15 which spread out the gas evenly and after having passed through the wall of the cone of the retort in the region 14 lead directly into the inside of such region 14 where the solids are dropped from the retaining tables of the discharging mechanism 13.

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As is to be seen from Figure 9 the cold gas injector nozzles 15 may merely consist of a chamfered pipe terminal with the cut part 15A turned inwards and of a size meant to prevent any gathering of particles upon the inside of the nozzle.

As can be easily seen by those skilled in the art, operation of the discharge mechanism 13, controlling the discharge of solids in the bottom of the cylindrical portion of the retort, governs the solids accumulation and the discharge of solids into the retort, as well as (not only because of the spaces between the retaining tables and the baffles, but also because of the loss of charge caused by the accumulated solids) controlling the upward flow of cold gases that come in through the nozzles 15. It should be understood that (a) such nozzles 15 emanate from external branches around the retort region 14 and their number will depend upon several factors, including the size of the retort, and that (b) such nozzles 15 enter the region 14 at points equiangularly spaced in a circular arrangement.

Such direct injection of gases without having to overcome the limitations imposed by the holes in the piping as in the previous system, enables a balance to be readily arrived at not only as regards the discharge of solids and gases but also as regards heat exchange, and reduces the need to compress the gases before they can enter into the bed of solids, which means a saving in both power and heat in general.

Solids that have just passed through the funnel-shaped region 14 will certainly be above 100°C when passing down the vertical duct 16 to the rejecting and sealing mechanism 17.

This latter mechanism consists essentially of one or more straight ducts of rectangular cross-section. Naturally according to whatever changes take place, and to the intensity of the flow of descending solids

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given off by the pyrolysis process occurring in the retort 9, there may be a need for more than one rejection and sealing mechanism 17 which will conform to branches of the descending duct 16 or to another duct that may have been adapted in the funnel-shaped bottom region 14 of the retort. However to facilitate understanding only one schematic description of the mechanism 17 will be given, as is shown in longitudinal section in Figure 1 as a sloping duct. The angle of the sloping duct 18 which represents the frame of the rejecting and sealing mechanism 17 is necessary in order to achieve the hydrostatic sealing of the retort and, in this case, its slope may be increased if the temperature and pressure conditions for the material under the process require it.

As shown in Figure 1 the rejecting mechanism consists of a rectangular cross-section sloping duct 18, housing an endless moving belt 19 running inside such duct 18 and supported by two pulleys 20 and 20A which also tension the belt so that it will be kept properly stretched and be driven by motors (not shown) applied to one of the pulley. The number and the arrangement of the pulleys is given merely as an example to help understand the invention, since the belt may use many arrangements of pulleys or tensioning means. From Figure 1 it can be seen that the outside of the moving belt 19 is provided with drag blades 21 which may be substantially rectangular in shape and may be slightly concave or slightly curved towards the direction of movement of the belt, and consequently in the direction of their own movement. The body of each drag blade may also be provided with openings to help entrain the solids by diminishing drag of the water bath wherever such blades are immersed therein in the course of their travel. The direction of rotation may vary since this depends on whether the belt faces left or right when viewed from the front, and it may move either clockwise or counter-clockwise. However the rotation of the driving pulley should be such that when solids drop

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from the duct 16 they should first of all be taken to the bottom 24 of the rejecting and sealing mechanism 17, from where they will be entrained by means of the blades upon the bottom wall of the sloping duct 18 up to a higher point of such duct from where they will be emptied to the outside through an opening 22. The disposal of the stream 23 of reject solids is not crucial to this invention though it is expected that a series of factors, such as the temperature of the solids when they come into the vertical duct 15 linking the funnel-shaped bottom region 14 of the retort to the delivery and sealing mechanism 17, and the speed at which the blades of the moving belt 19 entrain the solids, will ensure that the solids will have acquired the least possible quantity of water so as to enable them to be easily led to a dump or to a place where they undergo further treatment.

Though practice has shown that the pressure within the funnel-shaped bottom region 14 of the retort is low and is just enough to effect proper distribution of cold gases in said bottom region and to cause them to penetrate while rising within the bed of solids of the retort, the sealing of the downwardly extending duct 16 must nevertheless be as tight as possible, not only to prevent harmful gases from escaping into the atmosphere but also so that the interaction of gases, solids and sealing water will ensure that any matter harmful to the environment such as phenols, acids, and the more complex nitrogenated and sulphurated substances shall be dissolved or dispersed in the water. Next to the bottom end 24 of the rejecting and sealing mechanism 17 is a means of discharging the sealing water 27 whenever required at stopping or starting times. Also in the bottom end area of the rejecting and sealing mechanism 17 is the connecting point 29 of the line 99 which is meant to maintain the level of the sealing water in the rejection mechanism. Figure 1 shows such water supplied by a branch 64 of the line 99. Although not shown in Figure 1 this stream of water could if necessary be stripped of

impurities before being injected into the rejecting and sealing mechanism 17.

Also, within the upper end of the sloping body of 18 of the rejecting and sealing mechanism 17, there is a means 28 to vent and govern any steam or other vapours given off, when necessary.

As also shown in Figure 1, there is a given difference between the level 26 within the rejecting and sealing mechanism 17 and that within the vertical duct 16 in the bottom region 14 of the retort, this being the result of pressure exerted by cold gases at the nozzles 15, and such difference in level is a parameter employed in controlling the retorting operation.

Throughout the above description the path of the solids during the pyrolysis process has been described. This pyrolysis will be examined more closely in terms of retorting pyrobituminous shales which have a potential oil content of not less than 4% by weight (that is, the oil can be obtained by cheap hot treatment).

As can be seen in a general way from Figure 1 and in greater detail from Figure 4, there is in area III (Figure 4) a side opening connected to a duct 10 linking up the top of the retort with a cyclone separator 29 (Figure 1). Thus during the process in which pyrobituminous shale is being retorted within the particle size range and oil content referred to above the gases from this outlet 10 in the retort at a temperature of from about 140°C to 220°C, or preferably, from about 160°C and 180°C, and at a pressure of about 0.7 kPa to 7 kPa (gauge pressure), entrain a mist of liquid close to its dew-point. This mist is about 3% to 25% by weight of the gas stream, which also holds solid particles in a fine dusty state. There is then an initial separation process in the cyclone separator 29 where a part of the liquid mist (referred to herein as heavy oil) and most of the dusty matter, is held back while the output liquid travels down a line 31 to a storage vessel 32, from which it runs with its impurities along line 33 to

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pump 37 which pumps it along line 38 to an oil cleaning system. This oil cleaning system is not described since it is not part of this invention. The vaporized matter within the gaseous stream issuing from the cyclone separator 29 travels along line 30 to a heat regenerator 34 where its temperature is brought down to from about 130°C to about 160°C, or preferably to the range 130°C to 140°C, prior to being compressed later. The heat regenerator 34 is preferably a boiler to generate low pressure steam for use directly in the process or for recompression to the high pressure steam regime. Use of such a regenerator 34 raises the thermal efficiency of the system since it enables better use to be made of heat and cools down the gases on the suction side of the recirculating compressor.

Thus the gases from the heat regenerator 34 are led along duct 35 to one or more electrostatic precipitators 36 for purifying by more efficiently separating all the mist and dusty matter in the gaseous stream. It has been found in practice that the method of operation described herein produces a separating efficiency of 98 to 99.8%. In another design the purifying unit can be alternatively one or more gas scrubber columns which separate as efficiently as the electrostatic precipitator 36. In order not to avoid confusion, this alternative design is not shown in Figure 1, though it is to be understood that it would stand in the place vacated by the electrostatic precipitator or precipitators 36.

The gases from the electrostatic precipitators 36, or from the gas scrubber columns, are carried by ducts 39 to the recirculating compressor 40 where they are compressed to a pressure in the range of 41 kPa to 68 kPa (gauge pressure), which is enough to overcome any flow resistance along their recirculating path. Flow of such gases from the compressor 40 along the line 41 at a temperature of about 170°C to about 220°C divides at point 41 into four streams.

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The first stream is carried by line 43 to the heater 44 where gases are heated up to about 500°C - 600°C and is then taken along the line 45 to the hot gas injectors 11 inside the retort. This first heated gaseous stream is what is referred to herein for practical purposes as the "hot recycling" and also as "hot gases".

The second stream is led along line 81 to the heat regenerator 82 where it is cooled down to a temperature in the range of about 100°C to 130°C, and then carried by line 83 to point 84 where it splits into lines 85 and 86. This second gaseous stream is known by those skilled in the art and is here referred to as "cold gases". The branch 85 of the stream is injected into the bottom conical region 14 of the retort 9, by means of the injectors 15 so that the pressure in such region 14 shall be about 15 kPa to about 50 kPa (gauge pressure). The other branch of the cold gas stream flows along a line 86 which splits up into several secondary streams so as to enable the gases to be injected under pressure through the injection means 18C inside retainers 10C, as can be seen in Figure 7. Thus because of the pressure to which the stream of cold gases which travelling along line 86 is subjected, it not only circulates through the retainer 10C but also acts as a means of injecting part of the "cold gas" stream into the descending bed of solids inside the retort.

The third stream of gases from the compressor 40 is carried by pipe 46 to a heat regenerator 47 where it is cooled down to a temperature of about 90°C to about 110°C and then flows along line 48 to an air cooled until 49 where the steam and the light oil are largely condensed. From the air-cooled unit 49 the gaseous stream is carried by a pipe 50 to a spray tower 51 where condensation of the remaining water and oil (gas washing) is effected by means of sprays of recirculated retorting water pumped up to the spray tower 51 along line 61 which divides into lines 61A, 61B, 61C to the spraying terminals. It should be pointed out that there are not just three spraying devices but, rather, many of them; the

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number three having been chosen herein merely for the sake of simpler and clearer explanation.

It should also be noted that introduction of the air-cooled unit 49 is a major improvement as compared with the process of Brazilian Patent 7105857, as regards mass and energy balance in the process. Without such cooling the water brought into the spraying tower would have had a much higher heat charge which would have called for a greater flow of liquids along line 61 and through branches 61A, 61B, 61C; this would also have required more cooling fluid in the heat exchanger 60 cooling the stream of recycled water for line 61 and, if flow in such line were not enough to meet the heat demand in the condensing tower 51, cooling water might have had to be brought in from some external source which would have meant a more powerful pump than that required by the thermal demand of the process as described. The condensed output from the spray-tower 51 is carried along line 53 to the system of separators 54 and 56 arranged in series joined by the liquid carrying pipe 55. From the top of the spray tower 51 the gas output, also known as "retort gas" issues along line 52 at a temperature of about 25 to 40°C and is taken to a suitable treatment and purifying unit, and from there it goes on to further stages before being made use of commercially.

The fourth stream is the part of the compressed gas recycling through the duct 41A connected to a point downstream of the cyclone separator 29.

Liquids coming into the separator 54 undergo an initial separation therein for the purpose of securing circulating water to be reinjected into the spray tower 51 for condensation of the liquid and scrubbing of the gas output.

As will be explained in detail later, the water separated in the first separator 54 does not require much settling, since the output into line 57 is

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pumped by a pump 58 to a heat exchanger 60 along line 59, and is then after cooling, carried along line 61 to the spray tower 51 to contact the very stream from which it was derived, so any oil that may have been drawn into line 61 will return to the tower 51, thus allowing better contact with and a better rate of coacervation of the particles. This also saves time in the operating cycle and saves construction costs since the separator-settler 54 is bound to be smaller than the corresponding separator of Brazilian Patent 7105857. The floating oil from the separator-settler 54 travels along the overhead carrying duct 55 to the second separating-settler 56 for a more thorough separation of the light oil and water, the light oil being led along line 65 to pump 66 for pumping along line 67 for part of it to enter line 68 which will carry it to an oil purifying system not described herein, and the remainder to enter line 69 leading to a point where it will join up with another stream of heavy oil carried along line 78.

This latter heavy oil stream comes from the liquid separated out by the electrostatic precipitators 36 by way of line 73 to a storage vessel 74 before passing along line 75 to pump 76 which pumps it along line 77 to be branched at 79 into lines 78 and 71. The part which travels along line 78 may, if desired, be the flow to join up with the light oil pumped along line 69.

This oil mixture, also known as washing oil, is gathered from lines 69 and 78 and travels along line 80 to cyclone separator 29 where it will serve to wash the cyclone constantly so as to remove as much as possible of the heavy oil and impurities thereof, and then take it along carrier duct 31 to storage vessel 32, after which it will follow the route already described for final purification and use.

It should be pointed out that, if desired, part of the outflow from the pump 37 may (as shown in Figure 1) be led off from line 38 to line 70 and then join up with the line 80 carrying the cyclone washing oil to

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cyclone separator 29.

As in the case of the oil separated and settled elsewhere in the system, the purified oil from the electrostatic precipitator 36, or alternatively from the gas scrubbers, after having passed through the storage vessel 74 and after having been pumped by the pump 76 and bled off (if necessary) into line 78, is carried by line 71 to an external unit (not shown) where it will be employed.

Continuing the comparison with Brazilian Patent 7105857 it should be noted that the set of separating-settlers 54 and 56 arranged in series, represents a great step forward in this art and offers an overall saving under the process. In Brazilian Patent 7105857 just one large scale separator was provided for the output from the spray tower, where the water was withdrawn after a reasonably long period of residence, (this resulting from trying to separate as thoroughly as possible in a single operation the oil from the water under conditions which even required introducing water from outside to add to that needed for spraying and washing in the tower), which raised material requirements for the process and made less use of much more economical recycling, which would have enabled a balance to be more easily achieved for the process.

The aqueous phase withdrawn from the separator-settler 56, is conveyed by the line 62 to pump 63 which pumps it along the line 64 to the system that makes use of soluble by-products and deals with final disposal after purifying to prevent any pollution of the environment.

The retorting process which, in the present case deals specifically with pyrobituminous shales and, as regards the interior of the retort, amounts to the interaction of suitable crushed solids on a moving bed with gases derived from the retorting itself in a previously heated stream and another substantially cold one, (following the general retorting scheme described in Brazilian Patent 7105857), offers several improvements described above,

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thereby making the process cheaper and using better energy balance. Many engineering and cost problems met within the earlier patent have been overcome with the present process and apparatus and fresh design details have been submitted to 5 solve the problems.

For example, the "cold gases" are introduced into the bottom part of the retort, more precisely, into the bottom conical region 14, through inlet nozzles 15, and a part thereof by means 18C of the retainers 10C of the 10 controlled discharge mechanism 13 at a temperature of about 110°C to about 130°C, and so that the pressure in such region 14 is held at about 15 kPa to about 50 kPa.

In this region the "cold gases pass through the solids falling from the discharge mechanism after the solids 15 have already undergone the whole retorting process and have exchanged heat with the stream of cold gases at temperatures above the gas inlet temperature. Because of the pressure at which said hot gases are injected, and also because of the flow resistance of the column of water provided inside the 20 rejecting and sealing mechanism 17, they will flow up through the bed of solids, first of all passing through the controlled discharge mechanism 13, joining up with the part of "cold gases" entering at the retainers 10C, and continuing to flow upward throughout the length of the retort. As has 25 already been shown, the solids underwent heating from the "hot gases" injected into the system by injectors 11 thereby releasing organic matter in the pyrolysis treatment proper, and from the set of injectors 11 the hot solids will flow downwardly losing heat to the "cold gases" of the rising 30 stream so that when such "cold gases" have reached the hot gas injector system 11 the "cold gases" ought to have become heated up to a temperature just slightly below the inlet temperature of the "hot gases". In turn the solids that give 35 up their heat to the "cold gases", but are still warm, will reach the vertical duct 16 at the outlet of the discharge cone 14 of the retort, at a

-30-

temperature still above the boiling point of water, so that during the final rejecting operation their temperature will be reduced through contact with the water bath within the rejecting and sealing mechanism 17 and will create a small quantity of steam which is automatically added to the rising flow of "cold gases".

The "hot gases" entering through the injecting device 11 will be at a temperature of about 500°C to about 600°C, so that when mixed with the now heated "cold gases" they will be in a suitable state to bring about the pyrolysis of the crushed pyrobituminous shale. It should be mentioned that in practice any temperature measured in the area where the creation of pyrolysis products is at its highest will be close to 500°C, but it should be understood that the aim is not to keep to a given temperature for the reaction, constantly and strictly controlling it, but rather to introduce the "hot gases" at the stated temperature range in such a way that there will be a proper flow of pyrolysis material, since within the retorting area itself (as indeed throughout all of the retort) there is in fact a vertical temperature gradient and not only one constant temperature, throughout the whole bed. This is so because the shale at the charging mechanism is at the outside surrounding temperature which will depend on the prevailing climatic conditions and it will gradually undergo drying, a sort of preheating process, and then the actual retorting itself, while its temperature rises as it travels from area IV of the non-segregating distribution mechanism shown in Figure 4 towards the area where the "hot gas" injectors lie. The temperature of the solids will then fall from said "hot gas" injector point towards the bottom of the retort, as already explained.

The gaseous stream withdrawn from opening 10 (Figure 4) at the top of the retort 9 at area III of the non-segregating distribution mechanism 8 entrains with it liquid in a misty state close to its dew-point,

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and which is chiefly a mixture of light and heavy hydrocarbons plus more complex sulphurated and nitrogenated compounds as well as water vapour not only from the vaporization of the sealing water for the bottom rejecting and sealing mechanism 17 but also from the moisture in the shale due to the location where it was mined or due to the conditions under which it was stored prior to being processed. The gaseous stream consists largely of light hydrocarbons (rather than heavy ones), hydrogen sulphide, hydrogen, some carbon dioxide brought about by the breakdown of mineral carbonates, and also minute quantities of nitrogen and oxygen from any air held by the solids or arising out of the breakdown of components belonging to the mixture of products created.

Another factor typical of this new process, since it is a parameter connected with the movement and the compaction of the descending bed of solids as well as with the pressure of the injected gases, is the rate at which the gases rise through the retort, which varies from the bottom towards the top of the retort. Thus the gases at the bottom of the retort, where the column of crushed solids to be overcome will be at their highest depth both because of the geometry of the retort and because the temperature is lower, will move at a speed of about 0.40 m/s in action whilst the corresponding rate in the upper layers becomes close to 1.5 m/s in action.

Depending on the operating conditions, which are chiefly related to the quality of the raw material processed, and taking into account outlet moisture and temperature of gases, the mist joining the stream of products issuing from the top of the retort may range from about 3 to about 25% by weight thereof.

To illustrate the use of this process in a plant provided with all the above described apparatus, in terms of a retort whose main cylindrical portion has an inside diameter of 5.5m, as shown in Figure 1, values are given taken from two sample runs, referred to as runs 1 and 2.

To make it easier such values have been set out in a table and labelled (see the table below) in terms of the characteristics of: - the material charged; the chief operating conditions; the yield by weight from the runs, and the properties of the compound oil and of the gases obtained from the runs under regular laboratory analyses of petroleum products, quantity analysis of component elements, and gas phase chromatography.

It should be understood that data provided herein is merely that obtained with practical examples, and that such values in no way limit this invention.

TABLE 1

<u>VARIABLES</u>	<u>UNITS</u>	<u>RUN NO. 1</u>	<u>RUN NO. 2</u>
<b>1. PROPERTIES OF CHARGE</b>			
Particle size range mm		6.3-63.5	6.3-76.2
Moisture..... % weight		3.7	2.7
Fischer assay.....			
Oil..... % weight		7.6	9.1
Pyrolysis water.... % weight		1.2	1.4
Residue..... % weight		87.8	85.4
Gas + losses..... % weight		3.4	4.1
Total carbon..... % weight		12.9	15.6
Total hydrogen..... % weight		1.8	2.1
Sulphur..... % weight		4.6	5.4
Gross heating value		1450	1730
<b>2. OPERATING CONDITIONS</b>			
Retorting rate..... kg/h.m <sup>2</sup>		2653	2270
Pyrolysis temperature °C		483	488
Hot recycling temperature °C		549	564
Top of retort temperature °C		158	194
Bottom of retort temp. °C		249	241
Top of retort pressure kPa		2.2	1.9
Bottom of retort press. kPa		17.8	14.0

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-33-

## Recycle discharge/shale

discharge..... kg/kg      0.83      0.96

## 3. YIELDS ON FISCHER ASSAY

Oil yield..... %      96.4      101.4

Gas yield..... %      81.1      111.9

## 4. OIL PROPERTIES

Specific Gravity at 20°      0.924      0.940

Total carbon..... % weight      85.7      84.6

Hydrogen..... % weight      11.2      11.8

Sulphur..... % weight      1.2      1.4

Nitrogen..... % weight      0.8      1.1

Viscosity at 38°C.. cSt      17      43

at 54°C.. cSt      9      19

Pour point..... °C      -4      -18

## 5. GAS PROPERTIES

## Composition

H<sub>2</sub>S..... % vol      26.1      33.9O<sub>2</sub>..... % vol      0.1      0.1N<sub>2</sub>..... % vol      2.3      2.1

CO..... % vol      0.6      0.7

CO<sub>2</sub>..... % vol      3.7      2.9H<sub>2</sub>..... % vol      19.3      17.6

Methane..... % vol      19.5      21.9

Ethane..... % vol      6.3      6.3

Ethane..... % vol      2.5      2.3

Propane..... % vol      3.0      2.8

Propane..... % vol      2.9      2.8

Butanes..... % vol      1.2      1.1

Butanes..... % vol      2.8      2.8

C<sub>5+</sub>..... % vol      9.7      2.7

Molecular weight...      29.5      26.7

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THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. Apparatus for recovering oil, gas and by-products from material impregnated with hydrocarbons, comprising a pyrolysis retort; charging and sealing means to avoid escape of the retorting gases within the retort during charging of the retort with particulate material to be pyrolysed; discharge means for removing pyrolysed particles from the bottom of the retort while substantially avoiding escape of the retorting gases; means for injecting hot retorting gas into the retort for contact with the particulate solid material in the retort for pyrolysis thereof; means for introducing colder gas into the retort at a location below the hot gas introduction means; gas outlet means from the retort and leading to separating means for separating dust and released hydrocarbons and by-products from the gas leaving the retort; means for returning a first stream of gas from said separating means to the retort by way of said hot gas injector means; means for returning a second stream of said gas from the separating means to said colder gas injection means in the retort by way of a heat recovery unit; and means for directing a third stream of the separated gases from said retort to a location of use of the gases; wherein the said hot gas injector means comprise a bundle of mutually parallel pipes of polygonal cross-section, each pipe comprising an upper vertex defined by the angle of intersection of two roof plates which are joined to two parallel vertical side plates each joined at their lower ends to a floor of the pipe, each of said side plates including a row of gas discharge holes along the length of the pipe, in the upper portion of said side plate, the lower portion of each said roof plate being extended downwardly and outwardly beyond the said line of intersection with the adjacent side plate to create an overhang for protecting the gas discharge holes from impact by descending solid particles.

34

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2. Apparatus according to claim 1, wherein said row of gas discharge holes is just below the line of intersection between the side plate and the associated roof plate.

3. Apparatus according to claim 1, wherein the floor of each polygonal cross-section pipe includes a pair of inclined floor plates which extend from the lower ends of the side plates to meet at a bottom vertex of the pipe.

4. Apparatus according to claim 1, wherein the gas discharge holes are arranged such that the difference in the rate of discharge of gas between said hole and the next downstream said hole in a said row is from 1 to 5%.

5. Apparatus according to claim 1, wherein the difference in the rate of discharge from the first hole to the last hole in a said row is from 1 to 5%.

6. Apparatus according to any one of claims 1-5, wherein the means for injecting said colder gas comprise further pipes having ends which are each chamfered to provide a sharp end level with the top of that pipe when viewed from one side thereof, said sharp end leading into the interior of a funnel-shaped bottom region of the retort, and each of said further pipes being arranged around a circumferential wall of said funnel-shaped region.

7. Apparatus according to claim 1, wherein said separating means comprises a cyclone separator receiving the particle and hydrocarbon-laden gas from the retort and separating the hydrocarbons as liquid from the gas stream with residual dust and the mist of hydrocarbon material, and wherein the gas stream from the cyclone separator then divides into said first, second and third streams.

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8. Apparatus according to claim 7, including a duct connecting the cyclone separator to an electrostatic precipitator for further separation of the dust and mist from said gas, and including a water-cooled heat regenerator for cooling the gases from the cyclone separator by from 20°C to 60°C before said gases arrive at the inlet of the electrostatic precipitator.

9. Apparatus according to claim 1, and including means for compressing the separated gas from said gas separating means, said compressing means being effective to pressurize said first and second gas streams.

10. Apparatus according to claim 9, and further including a spray scrubbing tower supplied with gas by said compression means and further including a heat regenerator and an air-driven cooler between said compression means and said spray scrubbing tower.

11. Apparatus according to claim 10, and further including first and second separating settling vessels for receiving the liquid output from said spray scrubbing tower, said first separating settling vessel being connected directly to the spray scrubbing tower for separation of the discharged liquid into a mainly aqueous stream communicating with means for circulating it to the spray scrubbing tower, and an upper hydrocarbon stream communicating with means for transmitting it to the second separating settling vessel of which the upper outlet feeds a pump for returning the separated oil to said gas separating means and a lower outlet is connected to means for pumping the aqueous phase from said second separating settling vessel to waste.

12. Apparatus according to any one of claims 1-5 or 7-11, wherein said sealing means for preventing escape of the retorting gas during charging of the retort comprise first and second vertically spaced rotary seal systems each

36

comprising a vaned rotor sweeping the interior of a cylindrical stator whereby solid material entering the top of the first rotary seal system is swept towards an outlet at the bottom end thereof and falls from said outlet to the inlet end of said second rotary seal system to be swept by the rotor thereof towards a discharge opening at the bottom of said second rotary seal system for feeding the discharged solids to the retort, and means for injecting an inert gas into the solid material passing from said first rotary seal system to said second rotary seal system.

13. Apparatus according to claim 1, and including means defining a fourth gas stream from the gas output of said retort gas separating means and for recycling said fourth stream to a point upstream of a separating unit of said separating means.

14. Apparatus according to claim 13, wherein said separating unit comprises an electrostatic precipitator of said separating means connected downstream of a cyclone separator of said separating means.

15. Apparatus according to claim 14, including a heat regenerator between the gas outlet of the cyclone separator and the gas inlet to the electrostatic precipitator, said heat regenerator comprising a heat exchanger to generate low pressure steam.

16. Apparatus according to any one of claims 1-5, 7-11 or 13-15, wherein the spacings of said holes along the polygonal cross-section pipes of said hot gas injector means is at least four times the diameter of the largest particle expected in said retort bed.

17. A process for recovering oil, gas and by-products thereof from material impregnated with hydrocarbons, comprising introducing the hydrocarbon-impregnated material to a retort while avoiding escape of the retort gas from

within the retort and preventing ingress of oxygen into the retort at the point of entry of the incoming particulate material; introducing hot pyrolysing gas into the retort for contact with the particulate hydrocarbon-impregnated material therewithin; introducing a relatively colder gas into the retort below the level of introduction of the hot pyrolysing gas; discharging the pyrolysed particulate material from the base of the retort while excluding discharge of the retort gases with said discharge material; discharging the retort gases and separating therefrom dust and hydrocarbon mist discharged therewith; recovering the thus separated hydrocarbons; passing a first part of the gas from the retort gas separating operation to a spray scrubbing tower; spraying water into the spray scrubbing tower to wash hydrocarbons and dust from the gas which is then discharged for use; separating the water and hydrocarbons discharged from the spray scrubbing tower in first and second separating and settling stages in which the first separating and settling stage provides water for the spray-scrubbing operation and a hydrocarbon constituent which is further separated in the second separating and settling stage; and dividing the separated hydrocarbons from the second separating-settling stage into a first stream which is returned to the retort gas separating operation and a second stream which is delivered for commercial use; wherein the said first part of the gas from the retort gas separating operation is cooled before treatment in said spray scrubbing tower; and wherein a second part of the gas from the retort gas separating operation is cooled before return to the retort as the said relatively colder gas.

18. A process according to claim 17, wherein the retort gas separating operation is carried out in two stages by way of a cyclone separating first stage and an electrostatic precipitating second stage with cooling of the gaseous phase being carried out between the first and second separation stages, to reduce the temperature of the gaseous phase by from 20°C to 60°C.

19. A process according to claim 18, wherein the gas from the second separation stage is compressed before division into said first and second parts and wherein the inlet to the compression operation is at a temperature of from 130°C to 160°C.

20. A process according to claim 19, wherein the inlet temperature to the compression stage is from 130°C to 140°C.

21. A process according to claim 17, wherein cooling of the gaseous stream before arrival at the spray scrubbing tower is effected by way of a first heat recovery stage and a second air-operated cooling stage to bring the temperature of the gas down to substantially 90°C before inlet to the spray scrubbing tower.

22. A process according to any one of claims 17 to 21, wherein the pyrolysing particulate material in the retort is allowed to fall onto support surfaces from which it is scraped by a reciprocating scraper system, and wherein part of the cooled gas to form said relatively colder stream is returned to the retort by being injected into the bearings of the drive means for the reciprocating scraper mechanism.

23. A process according to any one of claims 17 to 21, wherein the pressure difference between the gas-sealing inlet to the retort and the particle-removing bottom of the retort is substantially 1.36 kPa.

24. A process according to any one of claims 17 to 21, wherein the particulate material to be pyrolysed is pyrobituminous shale crushed to have a particle size of substantially 0.32 cm to 15.24 cm, and wherein the particulate pyrobituminous shale entering the retort is introduced thereto by a non-segregating distribution action which separates the shale into two concentric zones in non-

1318273

segregated beds.

25. A process according to any one of claims 17 to 21, wherein the temperature of the hot gas entering the retort is from 500°C to 600°C and the relatively colder gases entering the retort below the level of hot gas introduction having a temperature of from 110°C to 180°C and at a gauge pressure of from 15 kPa to 50 kPa.

26. A process according to any one of claims 17 to 21, wherein the pressure in the top of the retort is from 0.7 kPa to 7 kPa.

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27. In a process for obtaining oil, gas and other products from pyrobituminous shales and other matter impregnated with hydrocarbons, which includes introducing crushed shale from a hopper downwardly into the upper portion of a retort through rotating charging devices having gas operated sealing means, contacting said crushed shale in the top portion of the retort with a stream of retort gases, injecting hot gases at an intermediate point of said retort at a temperature of about 500°C to about 600°C, introducing a stream of cold gases at the bottom of said retort at a temperature ranging between 110°C and 180°C and at a pressure of 15 kPa to 50 kPa (pressure gauge), discharging the crushed shale through a bottom sealing mechanism, removing gaseous retorted matter above the zone where the shale undergoes pyrolysis treatment, directing said gaseous retorted matter to a cyclone (29) for separation of heavy liquid components from a gaseous stream containing very fine solids in a mist, precipitating the very fine solids, compressing said gaseous stream, directing a first portion of said compressed gaseous stream through a heater (44) to raise the temperature to about 500° - 600°C for injection into said retort to start pyrolysis of the shale, directing a second portion of said compressed gaseous stream through a heat regenerator to reduce the temperature to about 110° - 130°C and injecting said stream into the retort through nozzles (15), directing a third portion of said compressed gaseous stream through cooling means to a spray tower (51) for separation of a gaseous component for extraction and a liquid component consisting primarily of water and heavy oil, and directing the liquid component to a decanting system,

the improvement comprising:

(a) supplying the liquid component comprised of water and heavy oil to first and second separating decanters (54 and 56) connected in series, removing the bottom watery layer from said first decanter to a circulating pump (58), pumping the bottom watery layer into said spray tower as a spray for condensation of

4 /

said liquid components of the gases transferring the top layer containing mainly oil by a pipe (55) to said second decanter (56), removing the bottom watery part to a pump (63) for disposal while supplying the oily portion to a pump (66) from which part of the oily portion is separated for reuse outside the process, and recirculating the remainder of the oily portion to said cyclone (29),

(b) cooling gases issuing from said cyclone (29) in a heat regenerator (34) to lower their thermal charge and passing the cooled gases through electrostatic precipitators (36) to a compressor so as to cause the temperature of the gases to drop by about 20° to 60°C in order to enter the compressor at a temperature of about 130° - 180°C,

(c) directing a portion of said compressed gaseous stream from the compressor through a heat regenerator (47) and an air operated cooler (49) in sequence so that the temperature may be brought down to about 90°C and supplying the cool compressed gas to the spray tower (51), and

(d) injecting a portion of said compressed gaseous stream from the compressor as a "cold gas" into the bottom of the retort whereby the difference in pressure between the charging devices and sealing means of the retort and the bottom portion of the retort is about 1.36 kPa.

28. A process as set forth in claim 27, wherein the rising gases travel through the retort from the point at which the cold gases are injected up to the zone at which the gases are withdrawn from the top of the retort at a rate which rises from about 0.4 m/s in the bottom portion up to about 1.5 m/s in the top portion of the retort.

29. A process as set forth in claim 27, wherein the pressure in the top of the retort is between about 0.7 kPa to about 7 kPa.

30. In an apparatus for obtaining oil, gas and other products from pyrobituminous shales and other matter impregnated with hydrocarbons, wherein the apparatus comprises:

a retort having an upper portion, an intermediate portion and a bottom portion,

hopper means for introducing crushed shale downwardly into said upper portion of said retort through a rotating charging device having gas operated sealing means,

first injection means for injecting hot gases into said intermediate portion of said retort at a temperature of about 500°C to about 600°C,

second injection means for injecting cold gases into said bottom portion of said retort at a temperature ranging between 110°C and 180°C,

bottom discharge means having water sealing means for discharging crushed shale from said retort,

cyclone means,

duct means connecting said cyclone means to said upper portion of said retort for removing gaseous retorted matter to said cyclone means for separation of heavy liquid components from a gaseous stream containing very fine solids in a mist,

precipitator means connected to said cyclone means for precipitating the very fine solids from said gaseous stream,

compressor means connected to said precipitator means for compressing said gaseous stream,

heater means connected between said compressor means and said first injection means for raising the temperature of a first portion of said gaseous stream to about 500°C - 600°C for injection into said intermediate portion of said retort to start pyrolysis of the shale,

heat regenerator means connected between said compressor means and said second injector means for reducing the temperature of a second portion of said gaseous stream to about 110°C - 130°C prior to injection of the second portion

1318273

(Claim 30 cont'd...)

of the gaseous stream into said bottom portion of said retort,

spray scrubbing tower means connected to said compressor means for receiving a third portion of said compressed gaseous stream for separation of a gaseous component and a liquid component consisting primarily of water and heavy oil, and

decanting means connected to said spray scrubbing tower means for receiving said liquid component;

the improvement comprising:

said first injector means being comprised of a plurality of parallel elongated hollow prismatic elements each having a hexagonal cross-section and including two elongated top plates connected to each other at an angle to form an elongated top vertex,

a pair of elongated parallel vertical side walls connected to lower edges of said two top plates respectively, with each side wall having a horizontal row of holes extending therethrough along the length of the side walls adjacent said top plates, said top plates extending downwardly and outwardly beyond said side walls to provide an overhang on each side to protect said holes from being directly struck by any descending solid particles, said holes having diameters related to each other such that upon passage of a gaseous stream under pressure through the prismatic elements the difference in discharge between a first and second of said holes is from 1 $\frac{1}{2}$  to 5 $\frac{1}{2}$  and lower walls connected to said side walls and connected to each other at an elongated bottom vertex;

said second gas injection means being comprised of a plurality of horizontally disposed circumferentially spaced apart pipes extending through a side wall of said retort and having downwardly and outwardly chamfered inner ends to prevent blockage of said pipes;

water cooled heat regenerator means connected between

44

1318273

said cyclone means and said precipitator means for reducing the temperature of gases issuing from said cyclone means by about 20°C to 60°C prior to entering said precipitator means;

heat regenerator means and air-worked cooler means connected in series between said compressor means and said spray scrubbing tower means; and

said decanting means being comprised of a first separating decanter having bottom liquid discharge means connected to pump means and conduit means for returning said liquid to said spray scrubbing tower means and upper discharge means connected to a second separating decanter to conduit means wherein said second separating decanter is provided with first outlet means connected to pump means for removing a watery portion and a second discharge means connected to a pump and conduit means for removing an oily portion.

31. An apparatus as set forth in claim 30 wherein the heat regenerator means disposed between said cyclone means and said precipitator means comprises an indirect heat exchanger for raising low pressure steam.

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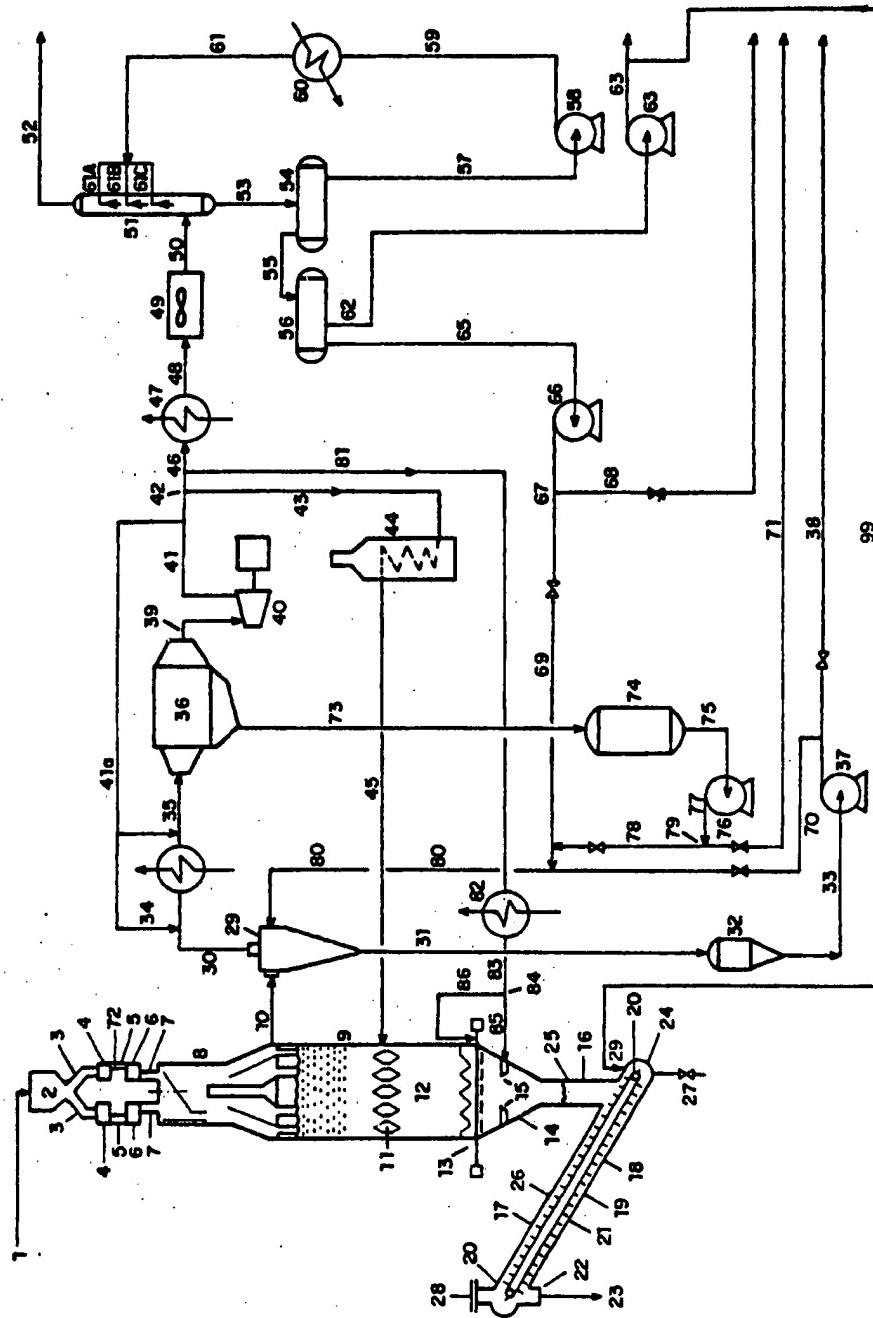


FIGURE 1

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7-2

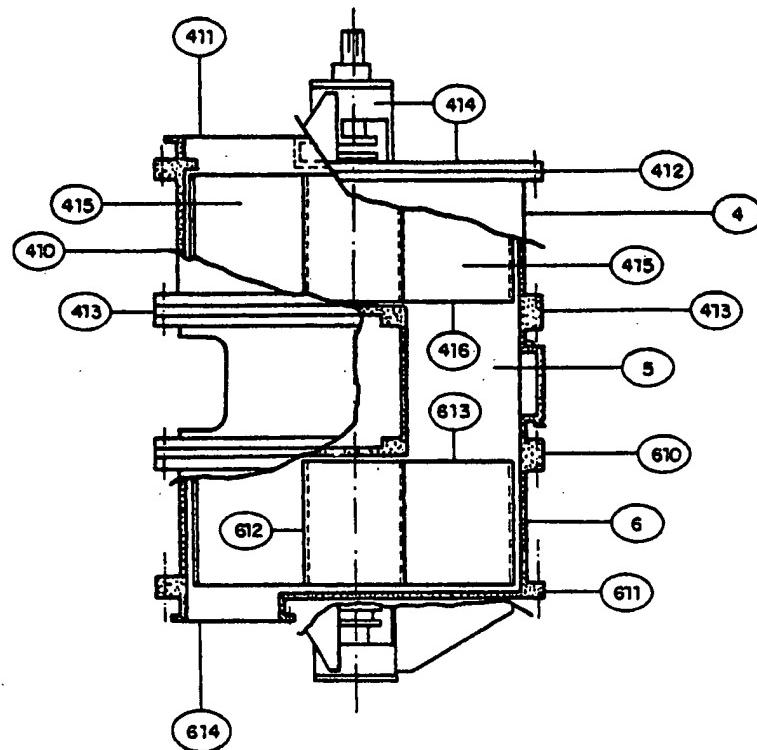


FIGURE 2

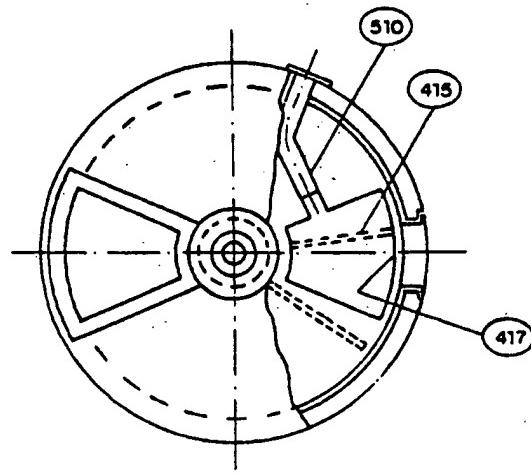


FIGURE 3

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7-3

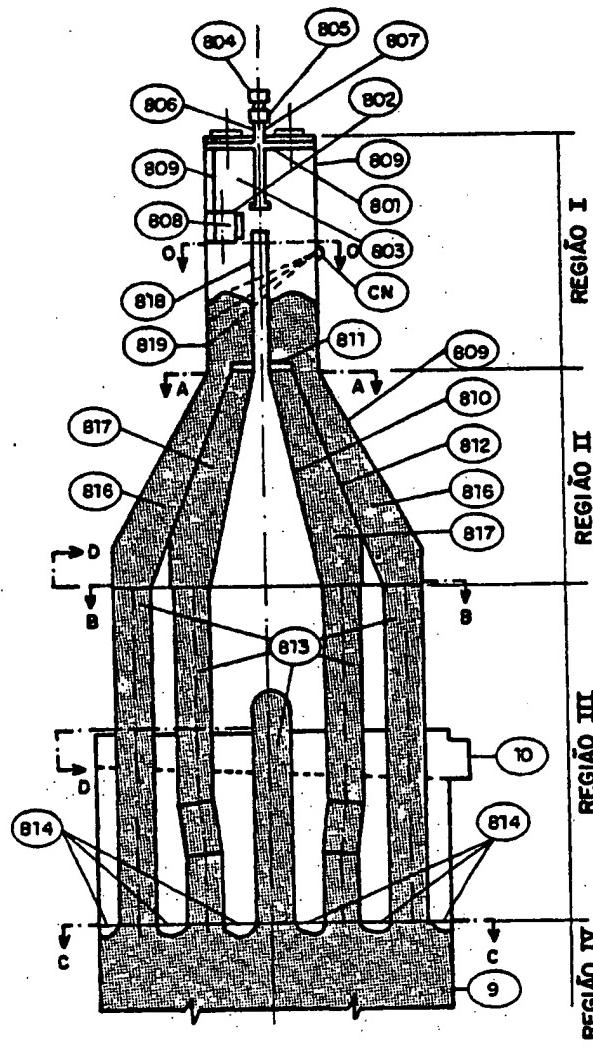


FIGURE 4

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7-4

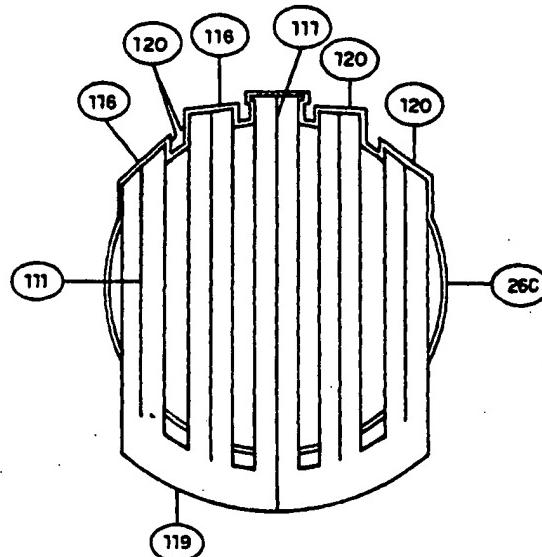


FIGURE 5a

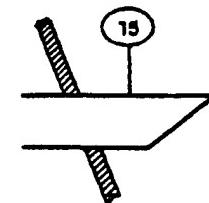


FIGURE 9

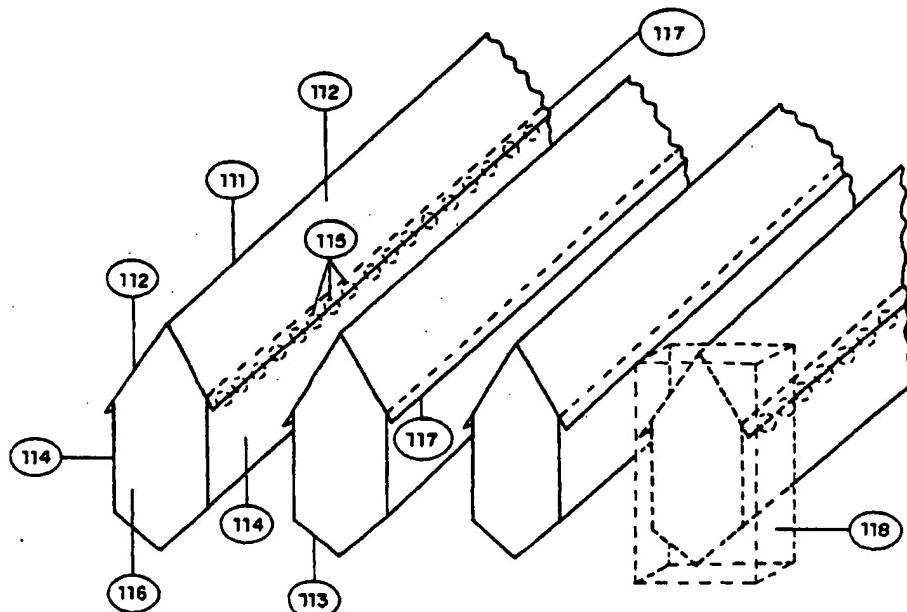


FIGURE 5

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1318273  
25

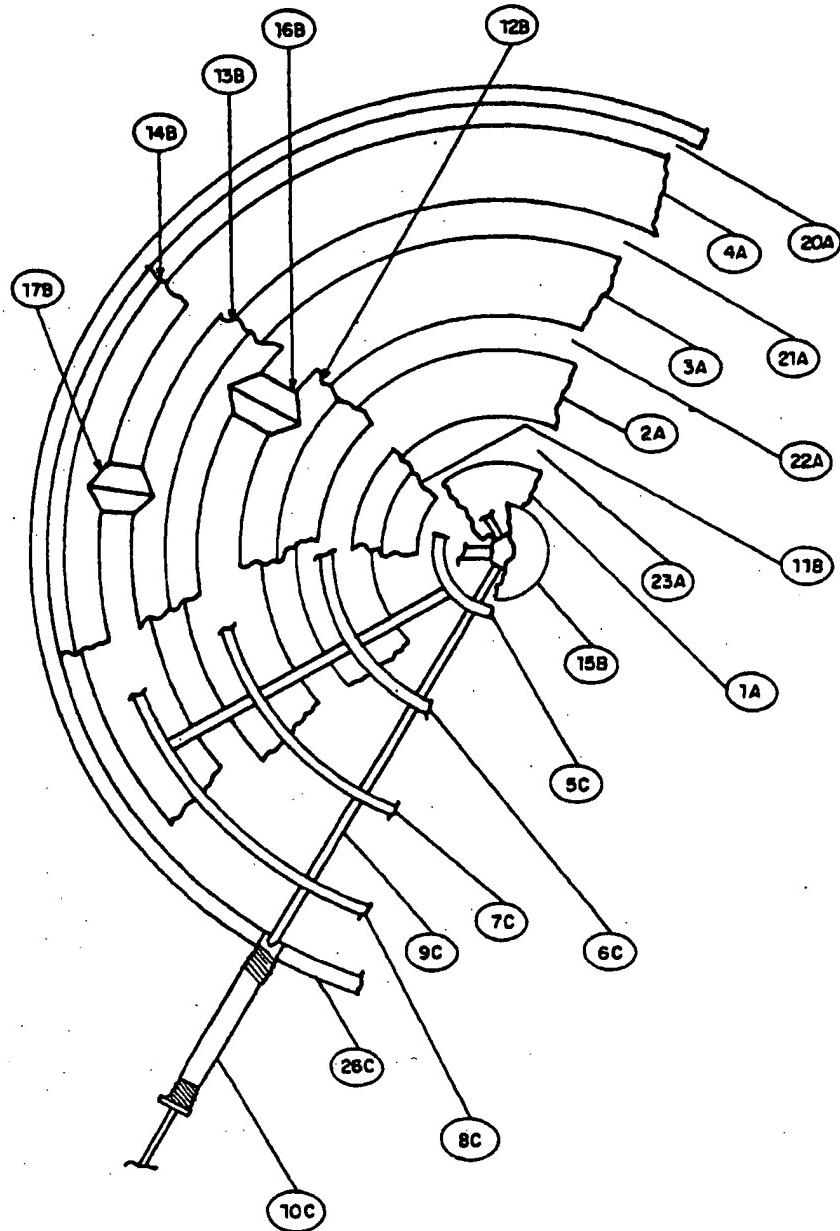


FIGURE 6

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1318273  
7-6

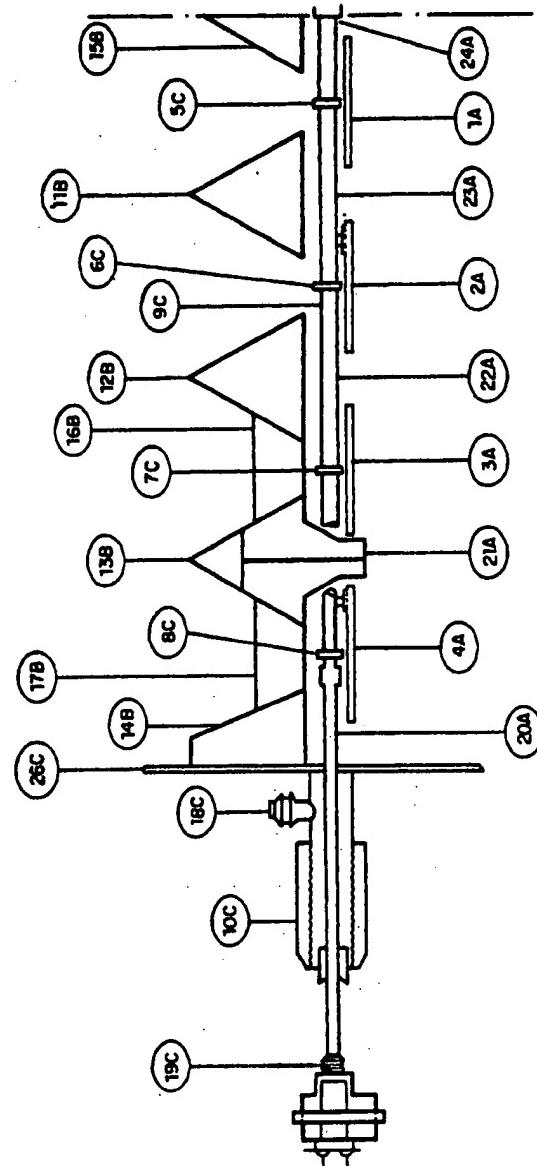


FIGURE 7

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7-7

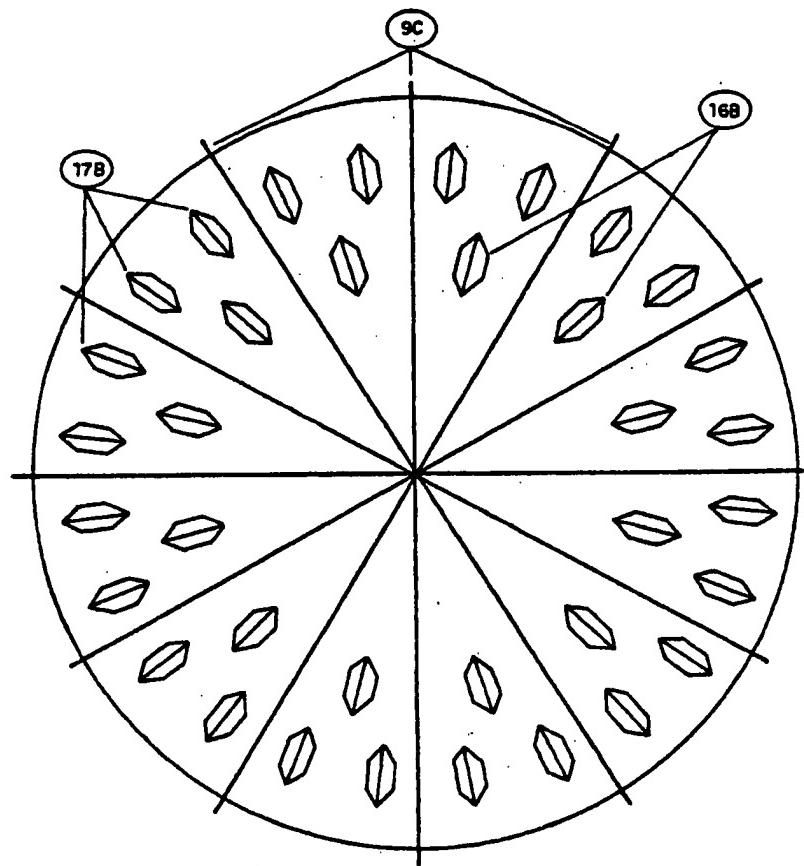


FIGURE 8

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